(19)日本国特許庁 (JP)

(12) 公開特許公報(A)

(11)特許出願公開番号

特開平5-238855

(43)公開日 平成5年(1993)9月17日

(51)Int.Cl. ⁵ C 0 4 B	41/87 41/80 41/87	識別記号 P A K T S	庁内整理番号 7038-4G 7038-4G 7038-4G 7038-4G 7038-4G	F I	技術表示箇所
-					審査請求 未請求 請求項の数 2(全 4 頁)
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(54)【発明の名称】 セラミックコーティング部材の製造方法

(57)【要約】

【目的】 セラミック基材とその被覆層の密着性が良好となり、高温強度、耐熱性に優れ、耐酸化性、耐食性が良好な、信頼性の高いセラミックコーティング部材の製造方法を提供する。

【構成】 基材の材料を炭化珪素、窒化珪素または炭化 珪素分散強化窒化珪素複合材から選択し、表面粗さとして十点平均粗さ(記号Rz)が 1.5μ m以上、かつ中心線平均粗さ(記号Ra)が 0.2μ m以上になる程度に基材の表面を機械加工、熱処理または化学的処理し、次いで基材の表面にアルミナ、ムライト、ジルコン、ジルコニア、イットリアから選択される少なくとも 1 種以上をプラズマ溶射する。プラズマ溶射により得られるセラミックコーティング層の層厚は 5.0μ m以上とする。

【特許請求の範囲】

【請求項1】 基材の材料を炭化珪素、窒化珪素または炭化珪素分散強化窒化珪素複合材から選択し、表面粗さとして十点平均粗さ(記号Rz)が1.5μm以上、かつ中心線平均粗さ(記号Ra)が0.2μm以上になる程度に基材の表面を機械加工、熱処理または化学的処理し、次いで基材の表面にアルミナ、ムライト、ジルコン、ジルコニア、イットリアから選択される少なくとも1種以上をプラズマ溶射することを特徴とするセラミックコーティング部材の製造方法。

【請求項2】 前記プラズマ溶射により得られるセラミックコーティング層の層厚を50μm以上とすることを特徴とする請求項1に記載のセラミックコーティング部材の製造方法。

【発明の詳細な説明】

[0001]

【産業上の利用分野】本発明はセラミックコーティング 部材の製造方法に関する。

[0002]

【従来の技術】従来より、高温で過酷な条件下で使用される高温構造材料としては、炭化珪素、窒化珪素等のセラミック材料が知られている。

[0003]

【発明が解決しようとする課題】しかしながら、このような炭化珪素あるいは窒化珪素等の高温耐熱材料といえども、これらの材料が高速の流速をもつ燃焼ガスに晒されると高温酸化、高温腐食等により部材が減肉されることが多い。このため、高速の流速をもつ燃焼ガスに晒される部材については、耐久性を向上するために耐酸化性を向上させることが最大の課題である。

【0004】この課題解決のため高温耐熱材料の基材表面に酸化物セラミックスを表面被覆する方法が提案されるが、この方法によると、基材の表面状態により酸化物セラミックスを被覆できなかったり、被覆できても高温での使用により短時間で剥離することがある。本発明の目的は、セラミック基材と被覆層の密着性が良好となり、高温強度、耐熱性に優れ、耐酸化性、耐食性が良好な、信頼性の高いセラミックコーティング部材の製造方法を提供することにある。

[0005]

【課題を解決するための手段】前記目的を達成するため の本発明によるセラミックコーティング部材の製造方法 は、基材の材料を炭化珪素、窒化珪素または炭化珪素分散強化窒化珪素複合材から選択し、表面粗さとして十点平均粗さ(記号Rz)が 1.5μ m以上、かつ中心線平均粗さ(記号Ra)が 0.2μ m以上になる程度に基材の表面を機械加工、熱処理または化学的処理し、次いで基材の表面にアルミナ、ムライト、ジルコン、ジルコニア、イットリアから選択される少なくとも 1 種以上をプラズマ溶射することを特徴とする。

【0006】前記十点平均粗さ(記号Rz)を1.5 μ m以上にし、かつ前記中心線平均粗さ(記号Ra)を0.2 μ m以上としたのは、これらの値未満であると、基材の表面にアルミナ、ムライト、ジルコン、ジルコンを強固に耐久性よく被覆することが困難になるからである。前記プラズマ溶射により得られるセラミックコーティング層の層厚は50 μ m以上としたのは、高速の燃焼ガス等に接触するような過酷な使用条件下では、前記セラミックコーティング層の層厚が50 μ m未満であると、長期間の使用における信頼性に問題があるからである。

[0007]

【作用】本発明のセラミックコーティング部材の製造方法によると、コーティング前のセラミック基材の表面を適正な面粗さにし、この粗面化された基材表面にプラズマ溶射による被覆層を形成するため、セラミック基材と被覆材の密着性が良好となり信頼性の高いセラミックコーティング部材を製作できる。

[0008]

【実施例】以下、本発明の実施例を説明する。 B_4 C、Cを添加物とする炭化珪素焼結体(実施例 $1\sim5$ 、比較例 $1\sim5$)、 Y_2 O_3 、Y b_2 O_3 を添加物とする窒化 珪素焼結体(実施例 $6\sim1$ O、比較例 $6\sim1$ O)、および Y_2 O_3 、Y b_2 O_3 を添加物とする窒化珪素を母材とし強化材として炭化珪素粒子あるいはウィスカーを添加した複合焼結体(実施例 1 $1\sim1$ 5)を基材に用い、基材表面粗さと酸化物被覆層の密着性について調べた。試験片形状は幅 4 mm、高さ 3 m、長さ 4 0 mmである。

【0009】詳細は下記表1に示す。

[0010]

【表1】

p						
区分基材		基材の表面 粗さ(μm)		溶射材	被復層の	耐熱サイクル
		Rz	Ra		付着状態	による密着性
実施例例例例例例例例例例例例例例例例例例例例例例例例例例例例例例例例例例例例	炭化珪素素 炭化珪素素 炭化珪素素 炭化珪素素 炭化珪素素素	1.7 1.7 1.7 1.7 1.6 1.6	0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2	アルミナ アムランフ ジルコニア マルシートリナ アルライト ジルン	良良良良良民好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好	良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良
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表1中、実施例1~5は、大気中、温度1400℃で1 0時間の熱処理を行った。実施例6~10は、70℃の HF中で1時間エッチング処理を行った。実施例11~ 15は、#36炭化珪素砥粒50wt%と#36アルミ ナ砥粒50wt%の混合砥粒でサンドブラスト処理を行った。

【0011】比較例1~5の基材は、炭化珪素で焼成面を#140ダイヤモンド砥石で機械加工した加工面である。比較例6~10の基材は、窒化珪素で加工後、大気中、温度1300℃で1時間熱処理を行った。比較例11~15の基材は複合材で加工後、大気中、温度1300℃で1時間熱処理を行った。前記実施例1~15および比較例1~15の基材に、アルミナ、ムライト、ジルコン、ジルコニア、イットリアの酸化物被覆層が約100μmの厚さになるようにプラズマ溶射により形成した。プラズマ溶射により形成された酸化物被覆層の付着状態および室温と1400℃間の加熱、冷却の繰り返しによる耐熱サイクル特性を測定評価した。その結果は表1に示すとおりである。

【0012】ィ 被覆テスト (酸化物被覆状態評価)

表1において、被覆層の付着状態は基材の表面に酸化物被覆層が均一に被覆されているかを目視により評価した。その結果、実施例1~15および比較例6~15については、酸化物被覆層の付着状態は良好であった。比較例1~5については酸化物被覆層が基材の一部にのみ付着しただけで被覆不能であった。基材の表面粗さが細かいと酸化物を被覆するのが困難であることが明らかとなった。

【0013】4 負荷テスト(耐熱サイクル評価) 基材と酸化物被覆の密着性は室温と1400℃との間の 加熱、冷却の繰り返しによる耐熱サイクルを行い、サイ クル1回毎に光学顕微鏡にて基材と被覆層界面を観察し た。その結果、実施例1~15は室温と1400℃との 間の20回の加熱、冷却の繰り返しでも基材と被覆層界 面には剥離が生じなかった。比較例6~15は室温と1 400℃との間の1回の加熱、冷却の繰り返しで基材と 被覆層界面に剥離が生じた。基材の表面粗さは適正な表 面粗さでないと耐熱サイクルに耐えられないことが明ら かとなった。

[0014]

【発明の効果】以上説明したように、本発明のセラミックコーティング部材の製造方法によれば、セラミック基材の表面を適正な面粗さにし、プラズマ溶射により基材の表面にアルミナ、ムライト、ジルコン、ジルコニア、イットリアから選択される少なくとも1種以上の被覆層

を形成するため、セラミック基材と酸化物被覆層の密着性および耐熱サイクル特性が良好になるので、高温で十分に耐え、高温での酸化性、腐食性が良好で信頼性の高いセラミックコーティング部材を製作できるという効果がある。

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PATENT ABSTRACTS OF JAPAN

(11) Publication number:

05-238855

(43) Date of publication of application: 17.09.1993

(51)Int.Cl.

CO4B 41/87 CO4B 41/80

(21)Application number: 04-043578

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(22)Date of filing:

28.02.1992

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(54) PRODUCTION OF CERAMIC COATING MEMBER

(57)Abstract:

PURPOSE: To provide a method of producing a ceramic coating member having good adhesion between a ceramic substrate and the coating layer, excellent high-temperature strength and heat resistance and good oxidation resistance and corrosion resistance and high in reliability. CONSTITUTION: The surface of a ceramic substrate consisting of a material selected from silicon carbide, silicon nitride or a silicon carbide-dispersed and strengthened silicon nitride composite material is subjected to mechanical processing, heat-treatment or chemical treatment so that ten point average roughness (Rz) as surface roughness may be ≥1.5μm and average roughness of center line (Ra) may be ≥0.2μm and then at least one kind of material selected from alumina, mullite, zircon, zirconia and yttria is subjected to plasma spray coating onto the surface of the substrate so as to give ≥50µm thickness of a ceramic coating layer to provide the objective ceramic coating member.

LEGAL STATUS

[Date of request for examination]

27.11.1998

[Date of sending the examiner's decision of

15.02.2001

rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection

[Date of requesting appeal against examiner's decision of rejection]

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CLAIMS

· [Claim(s)]

[Claim 1] The ingredient of a base material is chosen from silicon carbide, silicon nitride, or silicon carbide dispersion-strengthening silicon nitride composite. The ten-point average of roughness height (notation Rz) machines the front face of a base material as surface roughness to extent from which 1.5 micrometers or more and the center line average of roughness height (notation Ra) are set to 0.2 micrometers or more. The manufacture approach of the ceramic-coating member characterized by carrying out chemical preparation and carrying out the plasma metal spray of heat treatment or at least one or more sorts which are chosen from an alumina, a mullite, zircon, a zirconia, and yttria subsequently to the front face of a base material.

[Claim 2] The manufacture approach of the ceramic-coating member according to claim 1 characterized by setting to 50 micrometers or more thickness of the ceramic-coating layer obtained by said plasma metal spray.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the manufacture approach of a ceramic-coating member.

[0002]

[Description of the Prior Art] Conventionally, as an elevated-temperature structural material used under a severe condition at an elevated temperature, ceramic ingredients, such as silicon carbide and silicon nitride, are known.

[0003]

[Problem(s) to be Solved by the Invention] However, also although it is called elevated-temperature heat-resisting material, such as such silicon carbide or silicon nitride, if exposed to the combustion gas in which these ingredients have the high-speed rate of flow, high temperature oxidation, high temperature corrosion, etc. will thin down a member in many cases. For this reason, in order to improve endurance about the member exposed to combustion gas with the high-speed rate of flow, it is the biggest technical problem to raise oxidation resistance.

[0004] Although the approach of carrying out surface coating of the oxide ceramics to the base material front face of elevated-temperature heat-resisting material for this technical-problem solution is proposed, even if it cannot cover oxide ceramics with the surface state of a base material or can cover, according to this approach, it may exfoliate by use in an elevated temperature for a short time. The adhesion of a ceramic base material and an enveloping layer becomes good, the purpose of this invention is excellent in high temperature strength and thermal resistance, and oxidation resistance and corrosion resistance are to offer the manufacture approach of a good reliable ceramic-coating member.

[Means for Solving the Problem] The manufacture approach of the ceramic-coating member by this invention for attaining said purpose The ingredient of a base material is chosen from silicon carbide, silicon nitride, or silicon carbide dispersion-strengthening silicon nitride composite. The ten-point average of roughness height (notation Rz) machines the front face of a base material as surface roughness to extent from which 1.5 micrometers or more and the center line average of roughness height (notation Ra) are set to 0.2 micrometers or more. It is characterized by carrying out chemical preparation and carrying out the plasma metal spray of heat treatment or at least one or more sorts which are chosen from an alumina, a mullite, zircon, a zirconia, and yttria subsequently to the front face of a base material.

[0006] It is because it becomes difficult for that set said ten-point average of roughness height (notation Rz) to 1.5 micrometers or more, and under these values set said center line average of roughness height (notation Ra) to 0.2 micrometers or more to cover an alumina, a mullite, zircon, a zirconia, or yttria with sufficient endurance firmly on the surface of a base material. As for the thickness of the ceramic-coating layer obtained by said plasma metal spray, it is desirable to be referred to as 50 micrometers or more. Thickness of said ceramic-coating layer was set to 50 micrometers or more under a severe service

condition which contacts high-speed combustion gas etc. because a problem was in dependability [in / that the thickness of said ceramic-coating layer is less than 50 micrometers / prolonged use]. [0007]

[Function] Since according to the manufacture approach of the ceramic-coating member of this invention the front face of the ceramic base material before coating is made into proper field granularity and the enveloping layer by the plasma metal spray is formed in this base material front face by which surface roughening was carried out, the adhesion of a ceramic base material and a cladding material becomes good, and a reliable ceramic-coating member can be manufactured.

[0008]

[Example] Hereafter, the example of this invention is explained. The silicon carbide sintered compact which uses B4 C and C as an additive (examples 1-5, examples 1-5 of a comparison), Y2 O3 and Yb 2O3 The silicon nitride sintered compact used as an additive (examples 6-10, examples 6-10 of a comparison), And Y2 O3 and Yb 2O3 The compound sintered compact (examples 11-15, examples 11-15 of a comparison) which used as the base material the silicon nitride used as an additive, and added the silicon carbide particle or the whisker as reinforcement was used for the base material, and it investigated about the adhesion of base material surface roughness and an oxide enveloping layer. A test piece configuration is 40mm in width of face of 4mm, height of 3mm, and die length. [0009] It is shown in the following table 1 for details.

[0010]

[Table 1]

区分	基材		の表面 (μm) Ra	溶射材	被 復層 の 付着状態	耐熱サイクル による密着性
東実実実実実実実実実実実実実実実実実実実実実実実実実実実実実実実実実実実実	炭炭炭炭炭窒窒窒窒窒復複複複複化化化化化化化化化化化化化化化化化化合合合合合合合合合合合合	1.7 1.7 1.7 1.7 1.6 1.6 1.6 1.6 12.0 12.0 12.0 12.0	0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 2.2 2	アムジジイアムジジイアムジジイアムジジイアムジジイアムジジアカーシー・アー・アー・アー・アー・アー・アー・アー・アー・アー・アー・アー・アー・アー	段段好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好好	良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良良
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Examples 1-5 performed heat treatment of 10 hours at the temperature of 1400 degrees C among atmospheric air among Table 1. Examples 6-10 performed etching processing in 70-degree C HF for 1 hour. Examples 11-15 performed sandblasting processing with the mixed abrasive grain (#36 siliconcarbide abrasive grain 50wt% and #36 alumina abrasive grain 50wt%).

[0011] The base material of the examples 1-5 of a comparison is the processing side which machined the baking side by #140 diamond wheel with silicon carbide. The base material of the examples 6-10 of a comparison performed after processing and the inside of atmospheric air with silicon nitride, and performed heat treatment at the temperature of 1300 degrees C for 1 hour. The base material of the examples 11-15 of a comparison performed after processing and the inside of atmospheric air with composite, and performed heat treatment at the temperature of 1300 degrees C for 1 hour. It formed in the base material of said examples 1-15 and the examples 1-15 of a comparison by the plasma metal spray so that it might become the thickness whose oxide enveloping layer of an alumina, a mullite, zircon, a zirconia, and yttria is about 100 micrometers. Measurement evaluation of the adhesion condition of an oxide enveloping layer and room temperature which were formed of the plasma metal spray, and the heat-resistant cycle property by heating for 1400 degrees C and the repeat of cooling was carried out. The result is as being shown in Table 1.

[0012] ** Covering test (oxide covering condition evaluation)

In Table 1, as for the adhesion condition of an enveloping layer, the oxide enveloping layer evaluated by viewing whether it would be covered by homogeneity on the surface of the base material. Consequently, about examples 1-15 and the examples 6-15 of a comparison, the adhesion condition of an oxide enveloping layer was good. About the examples 1-5 of a comparison, covering only by the oxide enveloping layer having only adhered to some base materials was impossible. When the surface roughness of a base material was fine, it became clear that it is difficult to cover an oxide. [0013] ** Load test (heat-resistant cycle evaluation)

The adhesion of a base material and oxide covering performed the heat-resistant cycle by heating between a room temperature and 1400 degrees C, and the repeat of cooling, and observed the base material and the enveloping layer interface with the optical microscope for every one cycle. Consequently, in a base material and an enveloping layer interface, as for examples 1-15, exfoliation did not produce the repeat of 20 heating between a room temperature and 1400 degrees C, and cooling, either. Exfoliation produced the examples 6-15 of a comparison in the base material and the enveloping layer interface in the repeat of one heating between a room temperature and 1400 degrees C, and cooling. When the surface roughness of a base material was not proper surface roughness, it became clear [that a heat-resistant cycle cannot be borne].

[Effect of the Invention] As explained above, according to the manufacture approach of the ceramic-coating member of this invention In order to form at least one or more sorts of enveloping layers which make the front face of a ceramic base material proper field granularity, and are chosen from an alumina, a mullite, zircon, a zirconia, and yttria by the plasma metal spray on the surface of a base material, Since the adhesion and the heat-resistant cycle property of a ceramic base material and an oxide enveloping layer become good, it fully bears at an elevated temperature and is effective in the oxidizing quality in an elevated temperature and corrosive being good, and being able to manufacture a reliable ceramic-coating member.

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TECHNICAL FIELD

[Industrial Application] This invention relates to the manufacture approach of a ceramic-coating member.

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EFFECT OF THE INVENTION

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] However, also although it is called elevated-temperature heat-resisting material, such as such silicon carbide or silicon nitride, if exposed to the combustion gas in which these ingredients have the high-speed rate of flow, high temperature oxidation, high temperature corrosion, etc. will thin down a member in many cases. For this reason, in order to improve endurance about the member exposed to combustion gas with the high-speed rate of flow, it is the biggest technical problem to raise oxidation resistance.

[0004] Although the approach of carrying out surface coating of the oxide ceramics to the base material front face of elevated-temperature heat-resisting material for this technical-problem solution is proposed, even if it cannot cover oxide ceramics with the surface state of a base material or can cover, according to this approach, it may exfoliate by use in an elevated temperature for a short time. The adhesion of a ceramic base material and an enveloping layer becomes good, the purpose of this invention is excellent in high temperature strength and thermal resistance, and oxidation resistance and corrosion resistance are to offer the manufacture approach of a good reliable ceramic-coating member.

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OPERATION

[Function] Since according to the manufacture approach of the ceramic-coating member of this invention the front face of the ceramic base material before coating is made into proper field granularity and the enveloping layer by the plasma metal spray is formed in this base material front face by which surface roughening was carried out, the adhesion of a ceramic base material and a cladding material becomes good, and a reliable ceramic-coating member can be manufactured.

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EXAMPLE

[Example] Hereafter, the example of this invention is explained. The silicon carbide sintered compact which uses B4 C and C as an additive (examples 1-5, examples 1-5 of a comparison), Y2 O3 and Yb 2O3 The silicon nitride sintered compact used as an additive (examples 6-10, examples 6-10 of a comparison), And Y2 O3 and Yb 2O3 The compound sintered compact (examples 11-15, examples 11-15 of a comparison) which used as the base material the silicon nitride used as an additive, and added the silicon carbide particle or the whisker as reinforcement was used for the base material, and it investigated about the adhesion of base material surface roughness and an oxide enveloping layer. A test piece configuration is 40mm in width of face of 4mm, height of 3mm, and die length. [0009] It is shown in the following table 1 for details. [0010]

[Table 1]

		#++	7. 12. 12. 1	1		
区分	基材		の表面 (μm)	溶射材	被復層の	耐熱サイクル
ررط	2517	186	((2.11)	ומרני גים ו	付着状態	による密着性
		Rz	Ra		11/21/02	になる中国に
		<u> </u>				
実施例 1	炭化珪素	1.7	0.2	アルミナ	良好	良好
実施例 2	炭化珪素	1.7	0.2	ムライト	良好	良好
実施例 3	炭化珪素	1.7	0.2	ジルコン	良好	良好
実施例 4	炭化珪素	1.7	0.2	ジルコニア	良好	良好
実施例 5	炭化珪素	1.7	0.2	イットリア	良好	良好
実施例 6	室化珪素	1.6	0.2	アルミナ	良好	良好
実施例 7	窒化珪素	1.6	0.2	ムライト	良好	良好
実施例 8	室化珪素	1.6	0.2	ジルコン	良好	良好
実施例 9	室化珪素	1.6	0.2	ジルコニア	良好	良好
実施例10	窒化珪素	1.6	0. 2	イットリア	良好	良好
実施例11	複合材	12.0	2. 2	アルミナ	良好	良好
実施例12	複合材	12.0	2. 2	ムライト	良好	良好
実施例13	複合材	12.0	2. 2	ジルコン	良好	良好
実施例14	複合材	12.0	2. 2	ジルコニア	良好	良好
実施例15	複合材	12.0	2. 2	イットリア	良好	良好
比較例 1	炭化珪素	0.7	0.1	アルミナ	不良	_
比較例 2	炭化珪素	0.7	0.1	ムライト	不良	
比較例 3	炭化珪素	0.7	0.1	ジルコン	不良	
比較例 4	炭化珪素	0.7	0.1	ジルコニア	不良	
比較例 5	炭化珪素	0.7	0.1	イットリア	不良	_
比較例 6	窒化珪素	1.1	0.1	アルミナ	良好	不良 (剥離)
比較例 7	窒化珪素	1.1	0.1	ムライト	良好	不良 (剥離)
比較例 8	窒化珪素	1.1	0.1	ジルコン	良好	不良 (剥離)
比較例 9	窒化珪素	1.1	0.1	ジルコニア	良好	不良 (景雕)
比較例10	窒化珪素	1.1	0.1	イットリア	良好	不良 (剥離)
比較例11	複合材	1.1	0.1	アルミナ	良好	不良 (剥離)
比較例12	複合材	1.1	0.1	ムライト	良好	不良 (剥離)
比較例13	複合材	1.1	0.1	ジルコン	良好	不良 (剥離)
比較例14	複合材	1.1	0.1	ジルコニア	良好	不良 (剥離)
比較例15	複合材	1.1	0.1	イットリア	良好	不良 (剥離)

Examples 1-5 performed heat treatment of 10 hours at the temperature of 1400 degrees C among atmospheric air among Table 1. Examples 6-10 performed etching processing in 70-degree C HF for 1 hour. Examples 11-15 performed sandblasting processing with the mixed abrasive grain (#36 siliconcarbide abrasive grain 50wt% and #36 alumina abrasive grain 50wt%).

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